OIPE
SEP 0 8 2009
PATENT & TRADEMARK OFFICE

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	) PATENT
	)
Mohamed TAKHIM	) GROUP: 1793
	)
Serial No.: 10/583,344	) EXAMINER: QIAN, Yun
	)
Filed: August 4, 2006	) CONFIRMATION NO. 3133
	)
METHOD FOR ETCHING PHOSPHATE	) CUSTOMER NO. 25269
ORES	

* * * * * * * *

DECLARATION UNDER 37 C.F.R. §1.132

I, Marc Sonveaux, who resides at rue de la Vôte 36, B-5030 Gembloux, Belgium, hereby declare as follows:

1. I am a chemical engineer graduate of the Université Catholique de Louvain in Louvain la Neuve, Belgium, with 9 years of experience in research and development and industrialization in the chemical industry. I am the Research and Development Manager for EcoPhos sa in Louvain la Neuve, Belgium. EcoPhos sa is an organization which researches processes applicable to the phosphate industry.

2. I am familiar with the above-identified U.S. patent application and I have read the final Office Action of May 13, 2009, wherein the examiner has rejected claim 15 (the independent claim) and claims 2-5, 7, 9-11 and 14 (dependent claims) as being obvious over Hauge et al. (U.S. Patent No. 3,919,395). I am aware of the examiner's assertion that the change in sequence of neutralization in the present method compared

to that in Hauge et al. "would have been obvious to one of ordinary skill in the art absent evidence to the contrary."

3. I have conducted the following experimentation to demonstrate that the difference in neutralization steps is significant and unexpected, and certainly not obvious.

Comparison of Methods

According to the method of the present invention a preliminary neutralization is accomplished directly in the etching or digestion liquor. The filtrate is then separated by a first filtration (filtration 1). Thereafter the filtrate is submitted to a subsequent neutralization. DCP is precipitated and separated by a second filtration (filtration 2).

Hauge et al. disclose a process for digesting a phosphate rock using dilute HCl. The phosphate values are recovered from the solution in a two-stage specification with lime or ammonia. As noted in the final Office Action, page 4, last paragraph, the initially precipitated insolubles are separated by a first filtration (filtration 1). Thereafter a part of the impurities is precipitated by means of lime or ammonia and is separated by a second filtration (filtration 2). Finally, after a new addition of base, DCP is precipitated and separated by a third filtration (filtration 3).

These processes are diagrammed in Attachment A.

2

Experimentation

The two following experiments were carried out in order to compare both methods.

Present Method

A. Digestion of the rock

1. Mix 788 g water with 175 g HCl 33%, which results in a concentration of HCl of 6%.

2. Warm to 60°C and maintain at this temperature during the reaction.

3. Add 110g of a rock with 29% P_2O_5 and 32% Ca^{++}.

4. Agitate for 30 min.

5. Adjust the pH to 1.1 with a lime solution (preliminary neutralization).

6. Agitate for 30 min.

7. Filter the liquor (Filtration 1).

8. Analyze the obtained monocalciumphosphate (MCP) solution.

B. Production of DCP

1. Take 500 g of the filtrate of the previous step.

2. Adjust the pH to 3.2 with a lime solution (subsequent neutralization) and precipitate dicalciumphosphate (DCP).

3. Separate the DCP from the $CaCl_2$ solution by filtration (filtration 2).

4. Wash the obtained DCP with 1 X its mass of water.

5. Analyze the obtained DCP.

3

Hauge et al. Method

A. Digestion of the rock

1. Mix 788 g water with 175 g HCl 33%, which results in a concentration of HCl of 6%.

2. Warm to 60°C and maintain at this temperature during the reaction.

3. Add 110g of a rock with 29% P_2O_5 and 32% Ca^{++}.

4. Agitate for 30 min.

5. Filter the liquor (Filtration 1).

6. Adjust the pH to 1.1 with a lime solution (preliminary neutralization).

7. Agitate for 30 min.

8. Filter the slurry (Filtration 2)

9. Analyze the obtained monocalciumphosphate (MCP) solution.

B. Production of DCP

1. Take 500 g of the filtrate of the previous step.

2. Adjust the pH to 3.2 with a lime solution (subsequent neutralization) and precipitation of dicalciumphosphate (DCP).

3. Separate the DCP from the $CaCl_2$ solution by filtration (Filtration 3).

4. Wash the obtained DCP with 1 X its mass of water.

5. Analyze the obtained DCP.

Results

A. Digestion of the rock

The following table shows the data of the process and the obtained results at the end of the digestion step, i.e., after filtration 1 in the present method and after filtration 2 in the Hauge et al. method.

	Present Method	Hauge et al.	Units
Rock	110	110	g
Initial P_2O_5	31.8	31.8	g
Lime	35	-	g
Filtrate 1	1022	1041	g
[P_2O_5] Filtrate 1	2.8	2.9	%
Residue 1 (wet)	28.6	26.1	g
Humidity of residue 1	37.4	40.6	%
Yield 1 (of filtration 1)	90.7	95.0	%
Used MCP	-	963,0	g
Lime	-	14.7	g
[P_2O_5] Filtrate 2	-	2.9	%
Residue 2 (wet)	-	3.1	g
Humidity of residue 2	-	35	%
Yield 2 (of filtration 2)	-	89.3	%
Total yield of the digestion	90.7	84.8	%

It is also important to differentiate the chemical yield from the technical yield of the method. The chemical yield depends only on the reactions of digestion and of preliminary neutralization. The technical yield depends on the used devices. The above mentioned table shows the chemical yield. It is clear from the results that the present method is much better than the Hauge et al. method.

It is known that a technical yield of about 95% is obtained during a filtration step (loss on the filter, leakage, a.s.o.). Consequently, a

5

multiplication of the filtration steps diminishes this yield. It is also a great advantage of the present method to reduce the number of filtrations with respect to the Hauge et al. method.

It results also from the following tables that, on the one hand, the fluor content in the MCP solution (filtrate 1 in present method and filtrate 2 in the Hauge et al. method) is substantially reduced in the present method. The same remark is to do with the metallic impurities.

	MCP solution Present Method (filtrate 1) (ppm)	MCP solution Hauge et al. (filtrate 2) (ppm)
Fluor	379	846
Ag	0.03	0.03
Al	4.8	6.2
As	<0.5	<0.5
B	8.1	9.2
Ba	1.9	1.9
Be	0.17	0.18
Bi	<0.010	<0.010
Ca	33000	32000
Cd	1.4	1.4
Co	0.12	0.14
Cr	12	13
Cu	0.24	0.24
Fe	65	78
Hg	<0.010	<0.010
K	100	110
Li	0.12	0.2
Mg	1000	1000
Mn	3.1	2.7
Mo	0.19	0.25
Na	890	970
Ni	1.1	0.85
Pb	0.19	0.38
Sb	0.045	0.043
Se	<0.05	<0.05
Sn	0.067	0.032
Sr	210	210
Te	<0.01	<0.01
Ti	<0.5	<0.5
Tl	0.11	0.23

6

V	3.7	5.1
W	<0.010	<0.010
Zn	14	13

B. Production of DCP

The process for precipitating the DCP is identical in both methods.

The following table shows the data of the process and the obtained results at the end of the method, i.e., after filtration 2 in the present method and after filtration 3 in the Hauge et al. method.

	Present Method	Hauge et al.	Units
MCP solution	500	500	g
[P_2O_5] MCP solution	2.8	2.9	%
Lime	71.7	82.6	g
pH	3.2	3.2	
Obtained DCP (wet)	61	69.9	g
Obtained DCP (dry)	33.3	31.0	g
[P_2O_5] dry DCP	42	42	%
Humidity of DP	45.4	55.7	%
Yield of DCP precipitation	99.9	89.7	%

The data show that the DCP precipitation yield is substantially better according to the present method. The supply of lime is 15% lower for this step. The filtration process is more efficacious with the present method, while allowing a lower humidity of the DCP cake. This result is due to a better crystallization in the case of the present method.

The following tables illustrate the content of fluor and other metals in the obtained DCP.

7

	DCP Present Method (filtrate 2) (ppm)	DCP Hauge et al. (filtrate 3) (ppm)
Fluor	4287	8755
Ag	<0.05	<0.05
Al	190	170
As	1	1.3
B	3.2	6
Ba	3.5	6
Be	2.9	2.7
Bi	<0.05	<0.05
Ca	280000	280000
Cd	0.46	1.3
Co	1	1.5
Cr	180	170
Cu	0.51	0.55
Fe	920	1100
Hg	<0.010	<0.010
K	25	43
Li	0.076	0.18
Mg	220	500
Mn	23	41
Mo	3.6	3.9
Na	620	1200
Ni	1.1	0.97
Pb	5.2	6.9
Sb	0.58	0.65
Se	0.86	0.89
Sn	<0.05	<0.05
Sr	330	490
Te	<0.05	<0.05
Ti	3.9	2.9
Tl	<0.05	0.12
V	63	58
W	<0.05	0.07
Zn	170	200

The above indicated results show that the obtained purity of the produced DCP with the present method is substantially better than the purity according the Hauge et al. method.

Conclusions

8

The difference between both methods consists in the sequence

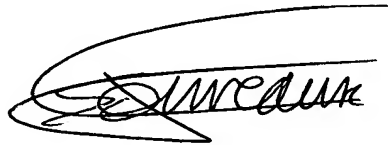| Present Method	Hauge et al.
1. Digestion with dilute HCl	1. Digestion with dilute HCl
2. Preliminary neutralization	2. Filtration 1
3. Filtration 1	3. Preliminary neutralization
4. Subsequent neutralization with DCP precipitation	4. Filtration 2
5. Filtration 2	5. Subsequent neutralization with DCP precipitation
	6. Filtration 3

This difference is essential because the properties of the process and the quality of the final product are greatly modified.

Advantage of the present method:

- Simpler : one step of filtration is abandoned and consequently less leakage.

- Yield of the reaction : 90.6% for present method (90.7% for the digestion and 99.9 % for the DCP production) versus 76% for Hauge et al. (84.8 % for the digestion and 89.7 % for the production of DCP).

- Better purity of MCP solution and DCP : -50% F and -30% cations.

It results from this comparison that a rock digestion with preneutralization without intermediary filtration is in an unexpected manner substantially more advantageous than a method with intermediary filtration after digestion and before preneutralizing.

9

I furthermore declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Marc Sonveaux

20/08/09

Date

10

Attachment A

Present Method

Hauge et al.